

## U.S. DEPARTMENT OF COMMERCE PATENT &amp; TRADEMARK OFFICE

B/O Form PTO-1390		<b>Transmittal Letter to the United States Designated/Elected Office (DO/EO/US) Concerning a Filing Under 35 USC 371</b>	Attorney's Docket Number SAND3002/REF/615
			U.S. Application Number (if known) <b>09/926050</b>
International Application Number PCT/GB00/00615	International Filing Date 22 February 2000	Priority Date Claimed 23 February 1999	
Title of Invention			
Applicant(s) for DO/EO/US			

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items under 35 USC 371:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 USC 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 USC 371.
3. ☒ This express request to begin national examination procedures (35 USC 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 USC 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed 35 USC 371(c)(2).
  - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 USC 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 USC 371(c)(3))
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 USC 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 USC 371(c)(4)). ( ☐ Executed ☐ Unexecuted )
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 USC 371(c)(5)).

Items 11 to 16 below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
  - ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☐ Other items or information:

Application Number (if Known) <b>09/926050</b>		International Application Number <b>PCT/GB00/00615</b>		Attorney's Docket Number <b>SAND3002/REF/615</b>	
				Calculations	PTO USE ONLY
17. The following fees are submitted: <b>Basic National Fee (37 CFR 1.492(a)(1)-(5)):</b> <input checked="" type="checkbox"/> Search report has been prepared by the EPO or JPO ..... \$860.00 <input type="checkbox"/> International Preliminary Examination Fee paid to USPTO (37 CFR 1.482) ..... \$690.00 <input type="checkbox"/> No International Preliminary Examination Fee paid to USPTO (37 CFR 1.482) but International Search Fee paid to USPTO (37 CFR 1.445(a)(2)) ..... \$710.00 <input type="checkbox"/> Neither International Preliminary Examination Fee (37 CFR 1.482) nor International Search Fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$1000.00 <input type="checkbox"/> International Preliminary Examination Fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$100.00				\$860.00	
<b>ENTER APPROPRIATE BASIC FEE AMOUNT</b>				<b>\$ 860.00</b>	
Surcharge of <b>\$130.00</b> for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).					
<b>CLAIMS</b>	<b>NUMBER FILED</b>	<b>NUMBER EXTRA</b>	<b>RATE</b>		
Total Claims	20      -20 =	0	× \$18.00	\$ 0.00	
Independent Claims	4      -3 =	1	× \$80.00	\$ 80.00	
Multiple Dependent Claims (if applicable)			+ \$270.00		
<b>TOTAL OF ABOVE CALCULATIONS</b>				<b>\$ 940.00</b>	
Reduction by ½ for filing by small entity, if applicable. Small Entity Status is asserted pursuant to 37 CFR 1.27 for this application.					
<b>SUBTOTAL</b>					
Processing fee of <b>\$130.00</b> for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).					
<b>TOTAL NATIONAL FEE</b>					
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). <b>\$40.00</b> per property.					
<b>TOTAL FEES ENCLOSED</b>				<b>\$ 940.00</b>	
				Refunded:	
				Charged:	

- a. ☒ A check in the amount of \$940.00 to cover the fees is enclosed.
- b. ☐ Please charge my **Deposit Account Number 02-0200** in the amount of \$ to cover the above fees.  
A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to **Deposit Account Number 02-0200**. A duplicate copy of this sheet is enclosed.

Note: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

**BACON & THOMAS, PLLC**  
 625 SLATERS LANE - FOURTH FLOOR  
 ALEXANDRIA, VIRGINIA 223124-1176  
 (703) 683-0500

DATE: August 21, 2001

Respectfully submitted,

*Richard E Fichter*

Richard E. Fichter  
 Attorney for Applicant  
 Registration Number: 26,382

## U.S. DEPARTMENT OF COMMERCE PATENT &amp; TRADEMARK OFFICE

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				Refunded:	
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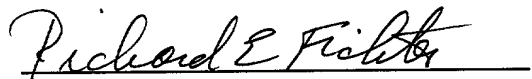
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DATE: August 21, 2001

Respectfully submitted,



Richard E. Fichter  
 Attorney for Applicant  
 Registration Number: 26,382

PATENT

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re application of:

SANDNES et al.

U.S. National Phase of PCT/GB00/00615

Entry papers filed herewith August 21, 2001

For: PROCESS FOR THE PRODUCTION OF A NUTRITIONAL COMPOSITION

Attention: PCT OFFICE

**PRELIMINARY AMENDMENT  
AND INFORMATION DISCLOSURE STATEMENT**

Assistant Commissioner for Patents  
Washington, D.C. 20231

Sir:

The present application is the U.S. national phase of international application number PCT/GB00/00615.

Please amend the above-identified application as follows:

**IN THE SPECIFICATION:**

Please add the attached ABSTRACT OF THE DISCLOSURE to the application.

**IN THE CLAIMS:**

Please replace claims 4-5, 7-8 and 11-15 with the following amended claims.

4(Amended). A process of claim 2 wherein the emulsion is heated by electromagnetic irradiation, preferably microwave irradiation.

5(Amended). A process of claim 1 wherein the emulsion is heated and dried by use of a disc drier.

7(Amended). A process of claim 2 comprising the steps of extruding or the emulsion into ropes, optionally degassing the emulsion, heating the formed emulsion to coagulate the protein therein, drying the coagulated emulsion, and chopping the dried emulsion onto pellets.

8(Amended). A process of claim 1 wherein the material to be emulsified comprises raw fish and gluten.

11(Amended). The feedstuff or nutritional composition of claim 9 further comprising a protein storage protein, preferably gluten.

12(Amended). The feedstuff or nutritional composition of claim 9 wherein the lipid content is 15 to 550 by weight on a dry weight basis.

13(Amended). The feedstuff or nutritional composition of claim 9 wherein the water content is 2 to 10% after drying.

14(Amended). The feedstuff or nutritional composition of claim 9 wherein the mixture is exposed to a partial vacuum after emulsification.

15(Amended). The feedstuff or nutritional composition of claim 9 wherein the final density is in excess of 1 g/ml.

**REMARKS**

Applicants have amended the claims in order to reduce the initial filing fee by deleting the multiple dependent claims from the application. Applicants retain the right to reintroduce any subject matter canceled by the present Amendment at any time during the prosecution of this application or any further application claiming benefit of this application.

In addition, an Abstract of the Disclosure has been added to the application.

Applicants are submitting herewith a copy of the Search Report which issued on International Application No. PCT/GB00/00615, of which the present application is the U.S. national phase. All of the publications cited in the International Search Report are listed on the attached Form PTO-1449. It is Applicants' understanding that, under the procedures of the PCT, copies of the cited publications will have been supplied to the U.S. Patent Office by the International Bureau. However, the Examiner is invited to contact the undersigned attorney if additional copies are necessary or would facilitate examination of the present application.


Otherwise, the Examiner is respectfully requested to return an initialed and dated copy of the attached Form PTO-1449 to confirm that all publications listed thereon have been considered and made officially of record in the file of this application.

Applicants understand that, under the procedures of the PCT, a copy of the priority document (GB 9904162.6, filed 23 February 1999) will have been supplied to the U.S. Patent Office pursuant to Rule 17 of the PCT Regulations. It is therefore respectfully requested that the first Official Action in the present application contain an indication that the appropriate priority document is in the file of this application.

In view of the above amendments, an early action on the application is now in order and is most respectfully requested.

Respectfully submitted,

BACON & THOMAS, PLLC

By:   
RICHARD E. FICHTER  
Registration No. 26,382

625 Slaters Lane - 4th Floor  
Alexandria, Virginia 22314  
Phone: (703) 683-0500  
Facsimile: (703) 683-1080

REF:kdd  
PA01.wpd

DATE: August 22, 2001



**Marked-Up Version Showing Changes Made**

**IN THE CLAIMS:**

Please replace claims 4-5, 7-8 and 11-15 with the following amended claims.

4(Amended). A process of claim[s] 2 [or 3] wherein the emulsion is heated by electromagnetic irradiation, preferably microwave irradiation.

5(Amended). A process of claim[s] 1 [or 2] wherein the emulsion is heated and dried by use of a disc drier.

7(Amended). A process of claim[s] 2 [to 4] comprising the steps of extruding or the emulsion into ropes, optionally degassing the emulsion, heating the formed emulsion to coagulate the protein therein, drying the coagulated emulsion, and chopping the dried emulsion onto pellets.

8(Amended). A process of [any of claims 1 to 7] claim 1 wherein the material to be emulsified comprises raw fish and gluten.

11(Amended). The feedstuff or nutritional composition of [either of claims 9 or 10] claim 9 further comprising a protein storage protein, preferably gluten.

12(Amended). The feedstuff or nutritional composition of [any one of claims 9 to 11] claim 9 wherein the lipid content is 15 to 550 by weight on a dry weight basis.

13(Amended). The feedstuff or nutritional composition of [any one of claims 9 to 12] claim 9 wherein the water content is 2 to 10% after drying.

14(Amended). The feedstuff or nutritional composition of [any one of claims 9 to 13] claim 9 wherein the mixture is exposed to a partial vacuum after emulsification.

15(Amended). The feedstuff or nutritional composition of [any one of claims 9 to 14] claim 9 wherein the final density is in excess of 1 g/ml.

## **ABSTRACT OF THE DISCLOSURE**

A process for the production of a nutritional composition, feed or feedstuff, comprising emulsifying a mixture containing raw fish, followed by heating and/or drying said emulsion.

3/pts

- 1 -

Process for the Production of a Nutritional Composition

The present invention relates to a process for the preparation of a fish-based fodder and to the fodder so prepared.

Aquaculture, sometimes referred to as fish farming, is a rapidly growing industry and provides an increasing demand for fish-feed, ie. fodder. Currently major components of fish-feed are marine proteins (in the form of fish meal) and marine oils (in the form of fish oils).

The term "feed" is generally used in the art to describe a product which meets the daily nutritional needs of the creature being fed with it, ie. it contains all the essential nutrients. The term "feedstuff" in comparison is used to refer to a component of the complete feed, e.g. a protein or fish oil or a component containing the necessary proteins and oils but without the proper vitamin content. As used herein, the term "nutritional composition" includes both complete feeds and feedstuffs.

In aquaculture, in particular salmon and catfish farming, fodder pellets are used as feed. These pellets are usually made from fish meal and fish oil and result in a more efficient use of the raw material. Thus for example 10 kg of capelin used directly as food for cod leads to generation of about 2 kg of cod corresponding to about 0.7 kg of cod fillets. If instead 10 kg of capelin is processed to produce fish meal and fish oil and used as fodder for farmed salmon, the yield is about 4.6 kg salmon or 2.8 kg salmon fillet. The energy yield moreover is significantly greater: the cod fillet corresponds to about 3 MJ while the salmon fillet corresponds to about 28 MJ.

The fish meal and the fish oil are produced by cooking the raw material (fish), and pressing the cooked

material to separate it into three fractions: water; fish oil; and protein. The protein fraction, the dried solid remnant of the cooking and pressing process, is about 70% protein, 10% fat and 10% water and is milled to produce fish meal. The oil fraction can be used directly for animal/fish feed production or alternatively may be purified and used for human consumption.

While it is termed fish oil, a more accurate term is perhaps lipid; both terms will be used below.

In the production of feed pellets, fish oil, optionally together with plant oils, is sprayed onto pellets formed from fish meal, optionally together with plant carbohydrates. In this way pellets with a lipid content of up to about 35% by weight can be produced. Ideally, the lipid content should be higher for optimal growth promotion in farmed fish such as salmon. However in warm temperatures, for example those experienced in summer, there is significant leakage of lipids from the feed pellets - a 500 kg sack might release as much as 30 to 50 kg of lipids. Not only is this wasteful of fish oil, since the released oil will not be consumed by the fish, but it results in the fodder being messy and difficult to handle, it results in clogging of automatic feeding systems (which often rely on pneumatic feed distribution) and it is environmentally undesirable as it puts oil onto the water surface.

Moreover, processing the raw material (fish) to separate out fish meal and fish oil and then recombining these to produce feed pellets involves a considerable usage of energy and equipment.

An alternative process for fish fodder production has been described in NO 903175 (Hamre). In this process the raw material, e.g. whole fish, fish heads, fish entrails, etc., is ground up, mixed with wheat meal, pelletized and then cooked in a microwave oven to produce pellets which float in water and which have a

moisture content of 10 to 30%. The microwave cooking coagulates the protein and prevents the pellets from coalescing - however the problem of lipid release is not overcome and the water content is undesirably high unless the pellets are to be used immediately. For storage-stable fish feed pellets, the water content is desirably below 10% by weight.

We have now found that improved feed and feedstuff, particularly in the form of pellets, can be produced if, before heating and/or drying, the raw mixture is emulsified, e.g. to a mayonnaise-like consistency. If this is done, the lipid content can be increased without lipid leakage problems and storage-stable pellets with a low water content can be produced.

Viewed from one aspect therefore the invention provides a process for the production of a nutritional composition, said process comprising emulsifying a material comprising raw fish and heating and/or drying the resulting emulsion, preferably heating said emulsion to coagulate the protein therein.

In the process of the invention, the raw fish used may be whole fish or parts of fish, e.g. entrails, heads, tails, etc., for example the waste material from fish filleting or gutting. Fish of the same species as the intended consumers of the nutritional composition are not recommended for use as the raw fish.

Besides the raw fish, other substances may be included in the material which is emulsified, e.g. fish meal, fish silage (hydrolysed fish), plant carbohydrate (e.g. wheat meal, corn meal, etc.), fish oil, plant oil, colouring agents, vitamins, minerals, pharmaceuticals (e.g. antibiotics, growth promoters, etc.), and plant proteins, especially storage proteins and most particularly gluten.

These additional substances may serve to provide a balanced diet for the creatures fed with the nutritional composition, e.g. the vitamins and minerals; they may

serve to adjust the lipid/protein balance, e.g. where the raw fish used is low in lipids, fish or plant oils may be used to increase lipid content; they may, like the colouring agents, be used to make the flesh of farmed fish more closely resemble that of wild fish, which is particularly desirable for farmed salmon; or they may serve to improve or protect the health of the creature receiving the feed, e.g. where antibiotics are used. The use of plant storage proteins, in particular gluten, however is especially desirable as it significantly and surprisingly improves the texture, physical strength and lipid retention ability of the product.

Thus with such additional substances included, the product of the process of the invention is in one preferred embodiment, a complete feed, especially a feed in pellet form or a feed or feedstuff in granular form (e.g. in powder, grain or meal form).

The material which is emulsified and heated should have a sufficiently high protein content to be coagulatable on heating. Typically the protein content will be 30 to 60% by weight, e.g. 35 to 55%, preferably 38 to 45%, most preferably about 40% on a dry weight basis. Of this, up to 100% may be fish protein, preferably at least 50% deriving from the raw fish. However up to 50% by weight of the protein may be plant protein, preferably gluten. Gluten especially preferably contributes 0 to 40%, e.g. 5 to 40%, more preferably 5 to 30%, e.g. 10 to 30% or 15 to 25%, and most preferably 10 to 20% by weight of the total protein. Gluten is preferably used as such, ie. as gluten rather than only in carbohydrate-containing wheat flour.

The high protein content of the material to be emulsified also serves to enhance the formation of the emulsion by serving as an emulsifying agent. In addition, this effect may optionally be enhanced by the

use of at least one specific emulsifier.

The mixture which is emulsified, and preferably coagulated, will preferably have a lipid content of 15 to 55% by weight on a dry weight basis, more preferably 20 to 40%. This may derive completely from the raw fish; however typically up to 25% of the total lipids may derive from added plant or fish oils. Suitable plant and fish oils include oils from cod, capelin, herring, sprat, blue whiting, sand eel, Norway pout, soy, oilseed rape, mustard seed, sunflower, safflower, etc.

Vitamins, colouring agents, pharmaceuticals and minerals will generally form only a minor portion of the mixture which is to be emulsified and coagulated, e.g. up to 10% by weight on a dry solids basis. Appropriate amounts can readily be calculated from the appropriate dosages and feed consumption rates for the creatures receiving the feedstuff.

Carbohydrates, e.g. digestible plant starch, for example wheat starch, will generally constitute up to 20% by weight on a dry weight basis of the mixture which is emulsified and cooked, preferably 5 to 15%.

The water content of the mixture to be emulsified and cooked will generally be in the range 40 to 75% by weight, for example 55 to 75% or 60 to 70%, but most preferably 45 to 60%. After cooking and drying, this will preferably be reduced to 0.5 to 70%, especially preferably 2 to 10% and more particularly 3 to 8% where the feedstuff is to be stored before use.

In the process of the invention, the mixture to be emulsified is preferably prepared by grinding up, chopping or mincing the raw fish, for example whole herring, sprat, mackerel or capelin, and then mixing in the extra substances, e.g. wheat starch, vitamin mix, gluten (e.g. from wheat and/or maize) and colouring agents (e.g. astaxanthin or cantaxanthin for salmon feed). This coarse mixture is then emulsified, e.g.



using a microcutter such as the Simo Microcutter MC250/115 PFVB175SS from Simo Industries A/S of Denmark.

In the Simo Microcutter, the mixture is fed at up to 6 tonne/ hour through die plates with 4 and 2.5 mm apertures and emulsified by rotating knife plates. The resulting emulsion contains oil droplets of about 1 to 50  $\mu\text{m}$  maximum dimension (e.g. diameter) and is substantially free of larger solid particles, ie. particles larger than 50  $\mu\text{m}$ , other than bone fragments which typically may be 200-500  $\mu\text{m}$ . Typically the proportion (e.g. by volume) of solid particles (other than bone fragments) larger than 5  $\mu\text{m}$  visible by light microscopy is less than that of oil droplets of this size or larger, for example by a factor of at least 10, more usually at least 100. As mentioned above, the emulsion typically has a mayonnaise-like consistency. Viewed by light microscopy this appears to have all or substantially all of the components of the mixture as a continuous aqueous phase or a discontinuous oil phase. This is readily distinguished from the pre-emulsification mixture produced by chopping and grinding in which solid particles deriving from the raw fish, in particular muscle fibres and large bone fragments, are a prominent feature and lipid droplets are larger than in the emulsion.

After emulsification, the mixture is preferably exposed to a partial vacuum (e.g. 0.1 to 0.9 bar) to reduce the amount of entrained gas. This avoids the production of a feed which will float in water as floating feeds are not desired by salt water fish farmers. Moreover this reduces the oxygen tension (ie. oxygen content) and thereby reduces oxidation of the lipids in the composition. For catfish farming however a floating feed is desirable and degassing may be omitted or performed less completely. The final feedstuff desirably has a density in excess of 0.6 g/mL, preferably in excess of 1 g/mL, more preferably in

excess of 1.2 g/mL.

In one embodiment of the invention, following emulsification, and if desired degassing, the emulsion is heated to coagulate the proteins and generate a lipid-retaining matrix. This may be done in several ways, e.g. by passage over a heated surface, by passage through a hot air dryer, by steam heating, by heating with electromagnetic radiation, by infra red heating, etc. However microwave heating is preferred.

In the heating step, the temperature and time of heating should be at least sufficient to coagulate the protein and create a matrix which encapsulates the lipid. It is not necessary that heating be effected such as to significantly reduce the water content of the mixture. In general, the mixture should be brought to a temperature in the range 50 to 100°C, preferably above 78°C.

The necessary extent of heating is readily determined in practice - with too little heating pellets of the emulsion are soft and deformable, and they stick together and coalesce. With sufficient heating, such pellets are self-supporting, transportable and non-coalescing. Too much heating is unproductive as it destroys protein quality and lowers the nutritional value of the feedstuff.

The heating step which coagulates the protein is preferably effected using electromagnetic (e.g. microwave) irradiation, e.g. at a frequency in the range 10 to 3000 MHz, preferably in the range 900 to 950 MHz. The irradiation intensity is preferably in the range 0.025 to 0.5 kW per kg/hour of emulsion throughput, especially 0.05 to 0.2 kW/kg.h<sup>-1</sup>, more especially 0.075 to 0.15 kW/kg.h<sup>-1</sup>. The use of microwave frequencies of 900 to 950 MHz, especially about 915 MHz is preferred to the use of higher frequencies due to the increased ability to penetrate the emulsion.

Desirably the water content is reduced as little as

possible during the coagulation step, with further drying to the desired final moisture content being carried out in subsequent treatment steps, e.g. using hot air drying. For dry, storage stable feed, the final moisture content is preferably less than 10% by weight; however for feed for use without storage, moisture contents of up to 30% are acceptable. Moisture content may be determined conventionally, e.g. using an infra-red moisture analyser such as a Mettler Toledo HR73 Halogen Moisture Analyser.

Before the coagulation step, the emulsion is preferably extruded or otherwise formed into sheets, or more preferably "ropes" of 2 to 40 mm thickness, especially ropes of 3 to 25 mm diameter. If desired the emulsion may be formed in "pellets"; however it is generally preferred to cut such ropes into pellets after coagulation. Such pellet sizes may be for example 2 to 30 mm, preferably 3 to 20 mm.

If extruded or otherwise formed as sheets, the coagulated emulsion may be broken into flakes, cut into strips or otherwise transformed into particles of the desired size.

Particularly desirably, the emulsion is formed, e.g. into sheets or ropes, before coagulation; coagulated; cut into pellets or strips or smaller sheets; dried on a perforated belt in a multi-sector hot air dryer; and if desired broken into flakes. Especially preferably the emulsion is extruded into ropes before coagulation, coagulated, cut into pellets and then dried on a perforated belt in a multisector hot air dryer. In one preferred embodiment, in an early sector of the multi-sector dryer, air flow is through the belt from below so as to separate the pellets while in a later sector air flow is through the belt from above so as to enhance the drying effect. Desirably a still later sector is arranged to cool the dried pellets.

The dryer used in this embodiment of the apparatus of the invention is conveniently a multi-sector dryer such as those produced by Lindauer Dornier GmbH, Lindau, Germany for drying of pelletized sewage sludge.

Where the product of the process of the invention is a feedstuff, a wide range of dryers may be used, e.g. multisector dryers as described above, hot air drum dryers, flash dryers, etc.

In an alternative embodiment of the invention, following emulsification, and if desired degassing, the emulsion is passed directly into a drying means, thus forming a nutritional composition in granular form (e.g. powder, grain or meal form).

The drying means may optionally be a high temperature drying means, for example a disc-dryer, or a lower temperature drying means, for example a vacuum dryer, spray dryer or flash dryer. For dry, storage stable feed-meal, the final moisture content is preferably less than 10% by weight; however for feed or feedstuff for use without storage, moisture contents of up to 30% are acceptable.

In one particularly preferred embodiment, the nutritional composition is a feedstuff produced in relatively fine grained form, e.g. pellets of 2 to 5 mm size, or in particular meal form, substantially free of plant carbohydrates and plant oils. This powdered or granulated feedstuff may then be used as an ingredient in the preparation of feed pellets by conventional methods, e.g. by pelletization or extrusion with binders and plant carbohydrates and addition of fish and/or plant oils and other substances as discussed above (e.g. vitamins, pharmaceuticals, colouring agents, etc). The addition of fish and plant oils may be performed so as to achieve a desired balance of fatty acid residues (e.g. of  $\omega$ -3 and  $\omega$ -6 acids) in the final product, which can be used as a human or animal (e.g. mammal, fish, reptile, etc.) feed or food supplement.

After drying and cooling, the nutritional composition may be packaged for storage or transport, e.g. in water-proof plastics containers such as sacks or drums.

The nutritional composition produced using the process of the invention is novel and forms a further aspect of the present invention. Viewed from this aspect the invention provides a nutritional composition produceable by emulsifying and coagulating and/or drying a mixture containing raw fish.

Viewed from a further aspect the invention provides a nutritional composition containing fish oil and fish protein, preferably coagulated fish protein, which is substantially free of muscle fibre fragments in excess of 200  $\mu\text{m}$  in length, preferably a gluten-containing composition.

The apparatus used in the process of the invention is also novel and forms a further aspect of the invention. Viewed from this aspect the invention provides apparatus for production of a nutritional composition, one embodiment of said apparatus comprising:

a grinder arranged to produce a ground raw fish mixture;

an emulsifier arranged to convert the ground raw fish mixture into an emulsion;

a heater arranged to coagulate the emulsion; and  
a dryer arranged to dry the coagulated emulsion.

Such apparatus preferably also comprises: a mixer to mix into the ground raw fish mixture other optional components such as vitamins, oils, minerals, gluten, starch, etc; a degasser to reduce the gas content of the emulsion; means for forming the emulsion into a desired form for coagulation in the heater, e.g. a spreader to produce sheets or an extruder to produce an emulsion extrudate; and a cutter to cut the coagulated emulsion into a desired form, e.g. a pelletizer to pelletize the

coagulated emulsion.

A further embodiment of the apparatus of the invention comprises:

a grinder arranged to produce a ground raw fish mixture;

an emulsifier arranged to convert the ground raw fish mixture into an emulsion and;

a dryer arranged to dry the emulsion.

Such apparatus preferably also comprises: a mixer to mix into the ground raw fish mixture other optional components such as vitamins, oils, minerals, gluten, starch, etc; a degasser to reduce the gas content of the emulsion.

Embodiments of the process, apparatus and products of the invention will now be described further by way of example and with reference to the following non-limiting Examples and to the accompanying drawings, in which:

Figure 1 is a schematic layout for an apparatus for the performance of the process of the invention;

Figures 2 and 3 are photomicrographs of emulsions used in the process of the invention; and

Figure 4 is a photomicrograph of a raw fish mixture that has been subjected to grinding and chopping but not emulsification.

Referring to Figure 1, there is shown an apparatus 1 for the performance of the process of the invention. Raw fish (e.g. whole herring) is transferred from receiving hopper 2 to a grinder 4 by screw feed 3. The ground fish is fed from grinder 4 to screw mixer 6 by screw feed 5. In screw mixer 6, a carbohydrate (wheat starch), pigment, gluten and vitamin mixture from hopper 7 is mixed with the ground fish and the resultant mixture is passed to a buffer mixer tank 8. The mixture from tank 8 is pumped by pump 9 into an emulsifier 10 where it is emulsified. The emulsion is passed into suction silo 11 and vacuum tank 12 where it is degassed at a pressure of 0.7 bar. The degassed emulsion is

pumped by pump 13 into extruder 14 which extrudes 12 mm diameter emulsion ropes onto a conveyor belt 15. Conveyor belt 15 transports the ropes of degassed emulsion through three heating sectors of a microwave oven 16 operating at 915 MHz. Water vapour from the oven is removed and condensed. The coagulated ropes leaving oven 16 are cut into pellets by cutter 17 and the pellets are transported into a seven-sector dryer 19 by belt 18. In the first three sectors of dryer 19, hot air is passed upwards through the perforated belt 20 carrying the pellets, and in the subsequent four sectors hot air is passed downwardly through the perforated belt. In a final sector, cool air is passed between the pellets and the dried and partly cooled pellets are subsequently sieved, cooled and bagged.

### Example 1 - formation of emulsion

The following materials are used to produce an emulsion for feeding to a microwave oven in an apparatus according to Figure 1:

Raw whole herring	100 parts by weight
Gluten	5.13 parts by weight
Wheat starch	4.30 parts by weight
Vitamin mixture	0.08 parts by weight
Colouring agent	0.02 parts by weight

Photomicrographs of an emulsion of raw fish and carbohydrate produced in this way are shown in Figures 2 and 3. As can be seen, the emulsion contains oil droplets of 1-50  $\mu\text{m}$  size. By way of comparison, from Figure 4 it may be seen that grinding and chopping raw fish, as in the procedure of Hamre (supra), does not produce such an emulsion.

The emulsion flow at 500 kg/hr is subjected to 75 kW microwave irradiation at 915 MHz, pelletized and dried to a moisture content of 7%. The resulting feed pellets show negligible lipid leakage.

### Example 2 - fat leak test

In order to demonstrate the stability of the fat in the product of the invention, the following pressure test was carried out.

Samples of crushed feed pellets formed by the process of the invention and of a well mixed blend of fish meal and fish oil were compressed in a specially designed test apparatus. Each sample consisted of 400g of material, with a fat content of approximately 43% of the dry mass. The following results were obtained:



		Pressure / Bar		
		4	8	17
Leakage from sample (g)	Crushed pellets	0	0	0
	Fish meal /oil mix	28	46	58

The results clearly show that the product of the invention gave no detectable fat leakage at pressures up to 17 bar, while the comparative mixture had lost more than 33% of its original fat content under the same conditions.

Claims

1. A process for the production of a nutritional composition, said process comprising emulsifying a material comprising raw fish and heating and/or drying the resulting emulsion.
2. A process of claim 1 wherein the emulsion is heated to coagulate the protein therein.
3. A process of claim 2 wherein the coagulated emulsion is further dried.
4. A process of claims 2 or 3 wherein the emulsion is heated by electromagnetic irradiation, preferably microwave irradiation.
5. A process of claims 1 or 2 wherein the emulsion is heated and dried by use of a disc drier.
6. A process of claim 1 wherein the drying is carried out using a belt drier, spray drier, flash drier or vacuum drier.
7. A process of claims 2 to 4 comprising the steps of extruding or the emulsion into ropes, optionally degassing the emulsion, heating the formed emulsion to coagulate the protein therein, drying the coagulated emulsion, and chopping the dried emulsion onto pellets.
8. A process of any of claims 1 to 7 wherein the material to be emulsified comprises raw fish and gluten.
9. A nutritional composition formed by emulsifying and coagulating and/or drying a mixture containing raw fish.

10. A nutritional composition containing fish oil and coagulated fish protein which is substantially free of muscle fibre fragments in excess of 200  $\mu\text{m}$  in length.
11. The feedstuff or nutritional composition of either of claims 9 or 10 further comprising a protein storage protein, preferably gluten.
12. The feedstuff or nutritional composition of any one of claims 9 to 11 wherein the lipid content is 15 to 55% by weight on a dry weight basis.
13. The feedstuff or nutritional composition of any one of claims 9 to 12 wherein the water content is 2 to 10% after drying.
14. The feedstuff or nutritional composition of any one of claims 9 to 13 wherein the mixture is exposed to a partial vacuum after emulsification.
15. The feedstuff or nutritional composition of any one of claims 9 to 14 wherein the final density is in excess of 1 g/ml.
16. An apparatus for production of a nutritional composition, said apparatus comprising:
  - a grinder arranged to produce a ground raw fish mixture;
  - an emulsifier arranged to convert the ground raw fish mixture into an emulsion;
  - a dryer arranged to dry the emulsion; and
  - optionally a heater arranged to coagulate the emulsion before it is dried.
17. The apparatus of claim 16 wherein the heating step is performed by electromagnetic irradiation, preferably microwave irradiation.

18. The apparatus of claim 16 wherein the heating and drying steps are performed by a disc drier or a hot air drier.

19. The apparatus of claim 16 wherein the drying step is performed using a hot air drier, a vacuum drier, a spray drier or a flash drier.

20. The apparatus of claim 16 wherein the emulsion is transferred directly to a disc drier, a hot air drier, a spray drier, a flash drier or a vacuum drier.

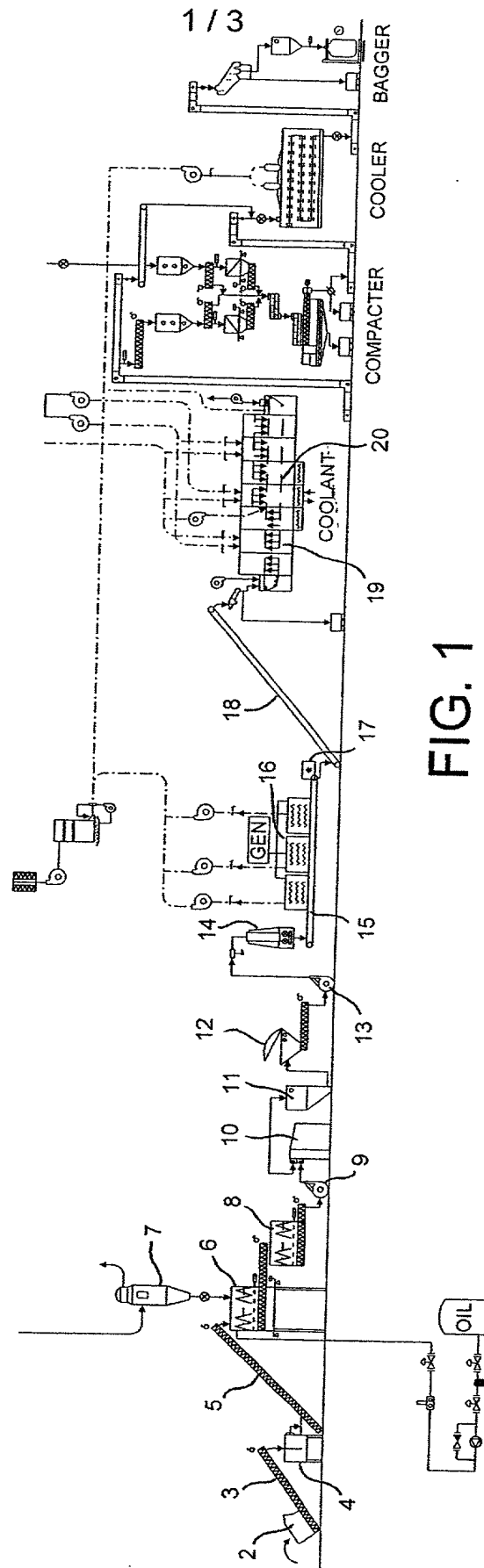
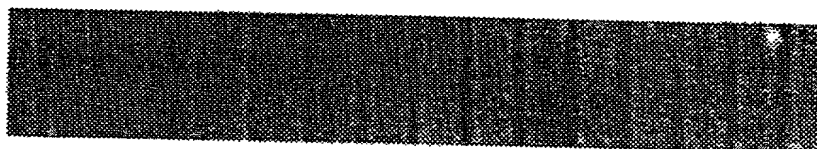
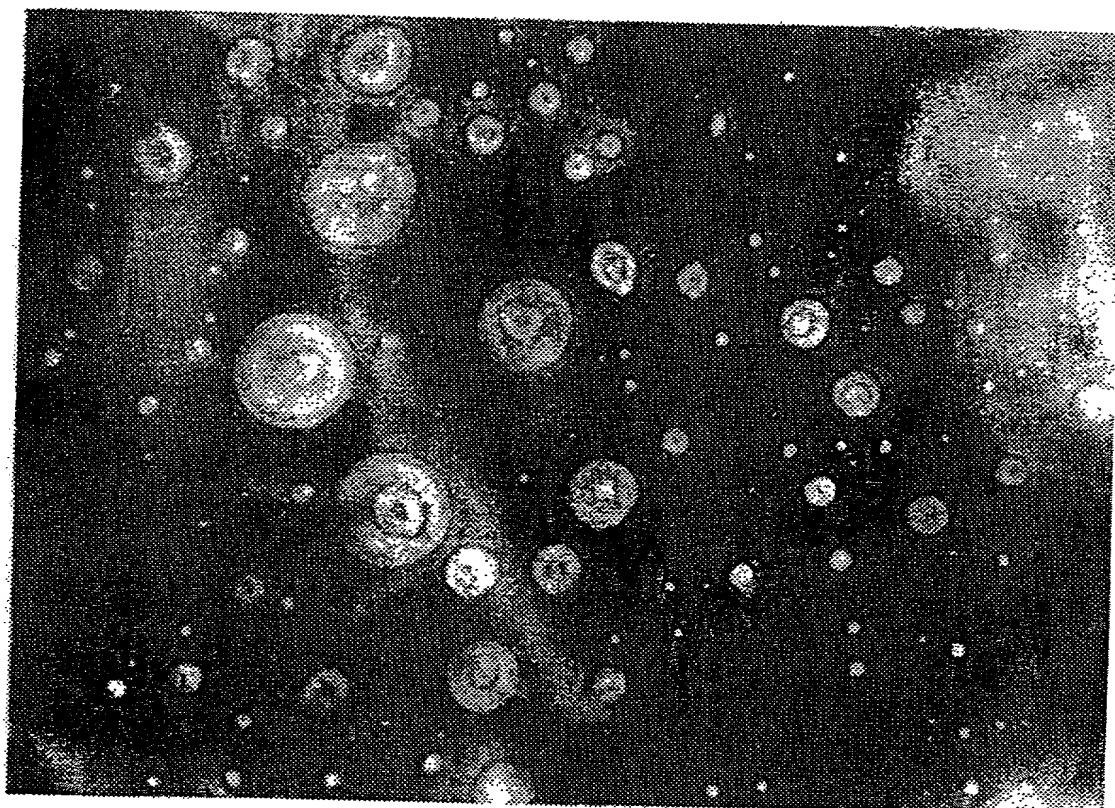


FIG. 1



0 50 100  $\mu\text{m}$

FIG. 2

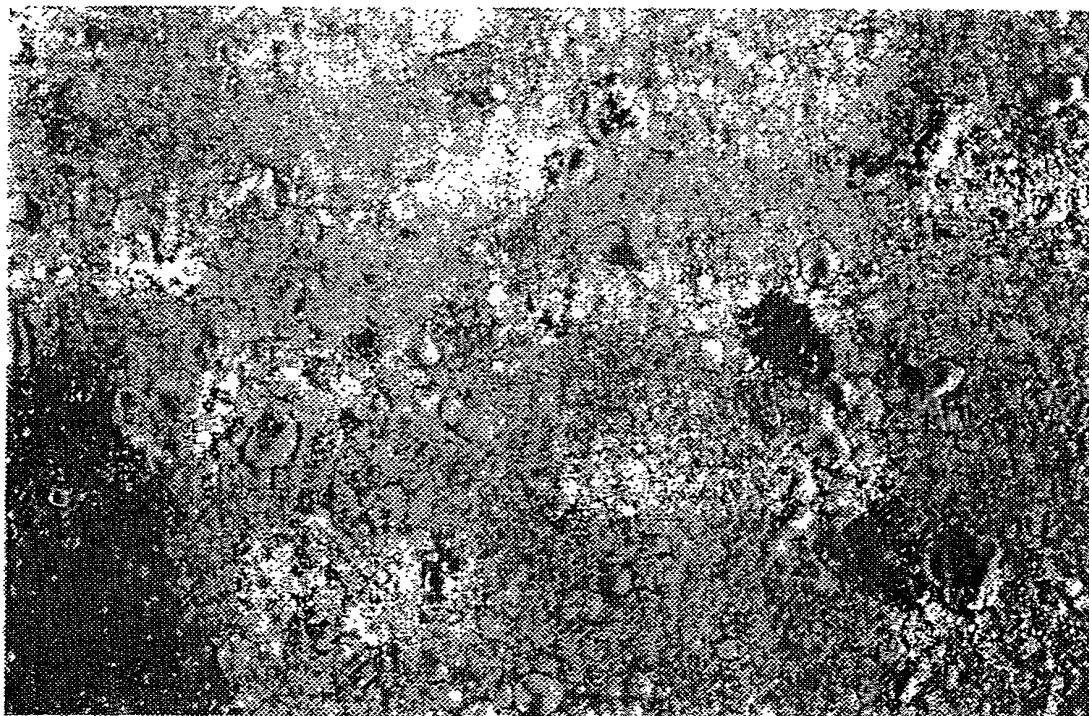


FIG. 3

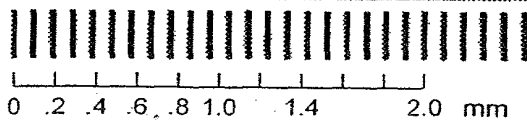
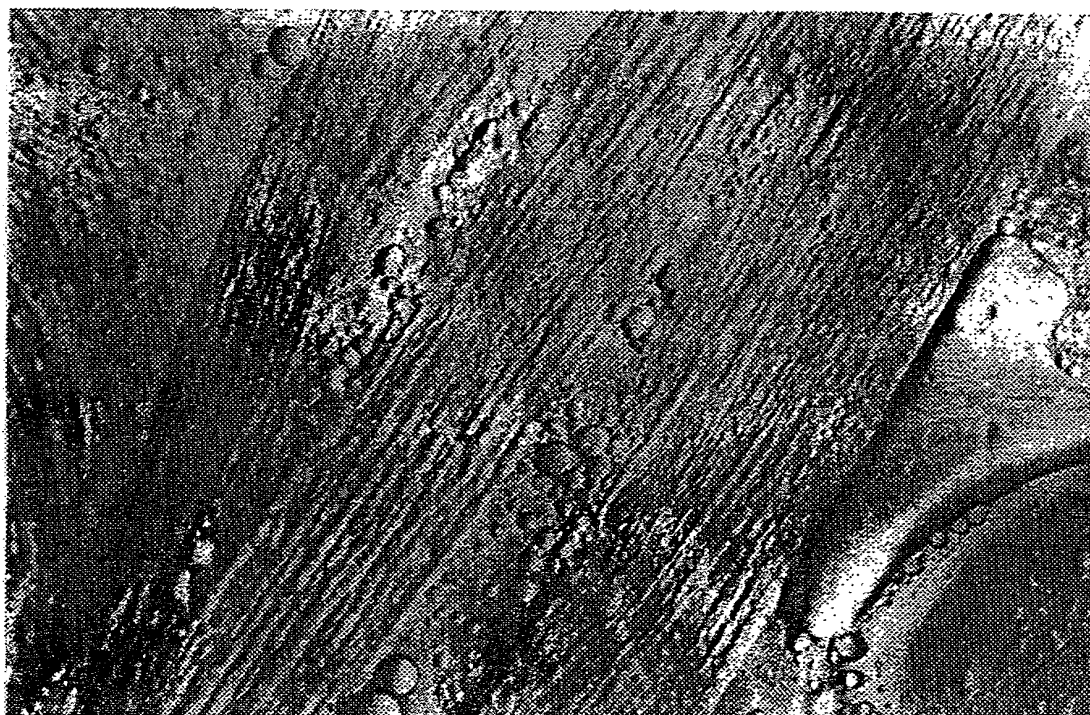


FIG. 4

**DECLARATION FOR PATENT APPLICATION AND APPOINTMENT OF ATTORNEY**

As a below named inventor, I hereby declare that my residence, post office address and citizenship are as stated below next to my name; I believe that I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention (Design, if applicable) entitled:

**PROCESS FOR THE PRODUCTION OF A NUTRITIONAL COMPOSITION**

the specification of which (check one):

☐ is attached hereto, or ☒ was filed on: **22 February 2000** as PCT International Application Number: **PCT/GB00/00615**

and (if applicable) was amended on:

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment(s) referred to above. I acknowledge the duty to disclose information which is material to patentability as defined in *Title 37, Code of Federal Regulations, §1.56*. I hereby claim foreign priority benefits under *Title 35, United States Code §119* of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

PRIOR FOREIGN APPLICATION(S)			PRIORITY CLAIMED	
Number	Country	Day/Month/Year Filed	Yes	No
9904162.6	Great Britain	23 February 1999	X	

☐ Additional Priority Application(s) Listed on Following Page(s)

**I HEREBY CLAIM THE BENEFIT UNDER TITLE 35 U.S. CODE §119(E) OF ANY U.S. PROVISIONAL APPLICATIONS LISTED BELOW.**

Application Number	Day/Month/Year Filed

☐ Additional Provisional Application(s) Listed on Following Page(s)

I hereby claim the benefit under *Title 35, United States Code, §120* of any United States application(s) or PCT international application(s) designating The United States of America listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of *Title 35, United States Code, §112*, I acknowledge the duty to disclose information which is material to patentability as defined in *Title 37, Code of Federal Regulations, §1.56* which became available between the filing date of the prior application(s) and the national or PCT international filing date of this application:

Application Number	Filing Date	Status - Patented, Pending or Abandoned

☐ Additional US/PCT Priority Application(s) listed on Following Page(s)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under *section 1001 of title 18 of the United States Code* and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

**POWER OF ATTORNEY:** I (We) hereby appoint as my (our) attorneys, with full powers of substitution and revocation, to prosecute this application and transact all business in the Patent and Trademark Office connected therewith: J. Ernest Kenney, Reg. No. 19,179; Eugene Mar, Reg. No. 25,893; Richard E. Fichter, Reg. No. 26,382; Thomas J. Moore, Reg. No. 28,974; Joseph DeBenedictis, Reg. No. 28,502; Benjamin E. Urcia, Reg. No. 33,805; and

I(we) authorize my(our) attorneys to accept and follow instructions from FRANK B. DEHN & CO. regarding any matter related to the preparation, examination, grant and maintenance of this application, any continuation, continuation-in-part or divisional based thereon, and any patent resulting therefrom, until I(we) or my(our) assigns withdraw this authorization in writing.

Send correspondence to: BACON & THOMAS  
625 Slaters Lane - 4th Floor  
Alexandria, VA 22314-1176

Telephone Calls to: (703) 683-0500

FULL NAME OF FIRST OR SOLE INVENTOR <b>Kjartan SANDNES</b>	CITIZENSHIP Norwegian <b>NOX</b>
RESIDENCE ADDRESS SeaGrain AS N-5392 Sorebu, Norway	POST OFFICE ADDRESS IS THE SAME AS RESIDENCE ADDRESS UNLESS OTHERWISE SHOWN BELOW <b>Kjartan Sandnes</b>
DATE <b>October 2, 2001</b>	SIGNATURE

☒ See following page(s) for additional joint inventors.



## CONTINUATION OF DECLARATION FOR PATENT APPLICATION AND APPOINTMENT OF ATTORNEY

Page 2

200 FULL NAME OF JOINT INVENTOR Bjorn MARKI	CITIZENSHIP Norwegian <i>NOX</i>
RESIDENCE ADDRESS Silfas, Scottsgata 3, Dreggen N-5023 Bergen, Norway	POST OFFICE ADDRESS IS THE SAME AS RESIDENCE ADDRESS UNLESS OTHERWISE SHOWN BELOW
DATE <i>October 2, 2001</i>	SIGNATURE <i>Bjorn Marki</i>

FULL NAME OF JOINT INVENTOR	CITIZENSHIP
RESIDENCE ADDRESS	POST OFFICE ADDRESS IS THE SAME AS RESIDENCE ADDRESS UNLESS OTHERWISE SHOWN BELOW
DATE	SIGNATURE

FULL NAME OF JOINT INVENTOR	CITIZENSHIP
RESIDENCE ADDRESS	POST OFFICE ADDRESS IS THE SAME AS RESIDENCE ADDRESS UNLESS OTHERWISE SHOWN BELOW
DATE	SIGNATURE

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DATE	SIGNATURE

☐ See following pages for additional joint inventors/priority applications.

(04AUG1998)